IS 5311 (1969): Code of safety for carbon tetrachloride
[CHD 8: Occupational Safety, Health and Chemical Hazards]

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IS : 5311 - 1969

Indian Standard
CODE OF SAFETY FOR CARBON TETRACHLORIDE

(Second Reprint FEBRUARY 1997)

UDC 661 723 24 614 8

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4 *February* 1970

AMENDMENT NO. 1 DECEMBER 2006
TO
IS 5311 : 1969 CODE OF SAFETY FOR CARBON TETRACHLORIDE

(Page 4, *clause* 3) — Insert the following new clause at the beginning and renumber the subsequent clause

'**3.1 General Information**

a)	Molecular formula	CCl_4
b)	Molecular weight	153 84
c)	CAS No	56-23-5
d)	UN No	1846'

(Page **4**, *clause* **4.1**. *last sentence)* — Substitute the following for the existing

'The Threshold limit values of carbon tetrachloride are TLV-TWA 5 ppm, TLV-STEL 10 ppm Carbon tetrachloride is suspected human carcinogen'

(Page 5, clause **4.1.1.2**) — Insert the following matter at the end

'Inhalation of high concentrations of carbon tetrachloride can have a narcotic effect Toxic if inhaled, ingested, or absorbed through the skin. Carbon tetrachloride primarily affects the liver and kidneys, but toxic effects in the lungs, and other tissues have been reported Overexposure can increase the risks of developing cancer '

(CHD 8)

Reprography Unit, BIS, New Delhi, India

Indian Standard
CODE OF SAFETY FOR CARBON TETRACHLORIDE

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(*Continued on page* 2)

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(*Continued from page* 1)

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Indian Standard

CODE OF SAFETY FOR CARBON TETRACHLORIDE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 July 1969, after the draft finalized by the Chemical Hazards Sectional Committee had been approved by the Chemical Division Council.

0 2 Carbon tetrachloride, afso known as tctrachloromethane contains 92 percent chlorine by weight. It is miscible with a large number of common organic liquids and is a powerful solvent for benzyl resins, chlorinated rubber, ethyl cellulose, asphalt, bitumen, fats, gums, rosin and waxes. The present principal use is in the manufacture of chloroflouromethanes used in aerosols and as refrigerants. Its other important applications are in fire extinguishing fluids and in cleaning and degreasing agents. It is also used as a component in formulations used for fumigating grains.

0.2.1 Its widespread application often without evidence of apparent ill effects, has resulted in under-rating its toxicity. Careful investigations have shown carbon tetrachloride to be one of the most harmful of the common solvents. A complete knowledge and understanding of the hazards of carbon tetrachloride is essential for safe handling in industry. The standard attempts to guide the users in the recognition of the hazards and in the recommended handling procedures.

0.3 In the preparation of this standard, considerable assistance has been derived from the Safety Data Sheet No. SD-3 for carbon tetrachloride issued by Manufacturing Chemists' Association, USA.

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1. SCOPE

1.1 This standard describes the properties of carbon tetrachloride, the nature of hazards associated with it and the essential information on storage, handling, packing, labelling, waste disposal, cleaning and repair of tanks, selection and training of personnel, personal protective equipment, and first aid.

1.1.1 This standard does not deal with specifications for design of buildings, chemical engineering plants, storage vessels and various safety equipment.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions' given in IS 4115-1966* and IS:4167-l966† shall apply.

3. PROPERTIES OF CARBON TETRACHLORIDE

3.1 Physical Properties — Some of the important physical properties of carbon tetrachloride are

a)	Physical state	Liquid
b)	Colour	Clear, colourless
c)	Odour	Ethercal odour similar to chlorinated solvents and clloroform
d)	Vapour density (air = I)	5 32
e)	Vapour pressure at 20 C	90 9 mm
f)	Vapour pressure at 30 C	143 0 mm
g)	Boiling point (760 mm Hg)	76 75 C
h)	Relative density, 25 C/4 C	1 584 5
k)	Solubility in water	Insoluble

3.2 Chemical and Hazardous Properties — Some important chemical and hazardous properties are

a)	Coriosivity	Dry carbon tetrachloride docs not corrode at normnal atmospheric temperatures In contact with water, particularly at elevated temperatures it corrodes iron and certain other metals
b)	Reactivity	Non-reactive and stable when dry. At temperatures above 250°C, moist carbon tetrachloride decomposes to form carbonyl chloride (phosgene), hydrochloric acid and other hydrocarbon products some of which are highly toxic.

4. HAZARDS ASSOCIATED WITH CARBON TETRACHLORIDE

4.1 Health Hazards — Carbon tetrachloride is toxic by inhalation and by ingestion. The principal hazard in the industrial use of carbon tetrachloride is from regular inhalation of small quantities of vapour. The toxic effects of carbon tetrachloride are markedly increased by intake of alcohol. The threshold limit value, accepted at present, of carbon

*Glossary of terms relating to chemical and radiation hazards and hazardous chemicals.

†Glossary of terms relating to air pollution

tetrachloride for a daily 8 hour exposure is 10 parts per million by volume (65 mg/m^3) in air.

4.1.1 *Acute Toxicity*

4.1.1.1 *Local effects* — Skin contact with the liquid carbon tetrachloride leads to dryness through the removal of natural oils from the skin. Repeated or prolonged exposure may cause dermatitis, cracking of the skin and danger of secondary infections. Eye contamination by liquid carbon tetrachloride causes burning, intense irritation and other symptoms of inflammation.

4.1.1.2 *Systemic effects* — The effects of excessive exposure of carbon tetrachloride may be both immediate and delayed. The immediate effects may include headache, and symptoms resembling inebriation or drowsiness and abdominal discomfort. The delayed effects may include severe damage to the liver and kidneys which may not become evident until 1 to 10 days after the exposure.

4.1.2 *Chronic Toxicity* — Subacute or chronic carbon tetrachloride poisoning may result from prolonged or repeated exposure to the vapours or the liquid.

4.2 Fire and Explosion Hazards — Carbon tetrachloride is nonflammable and non-explosive in air at ordinary temperature and pressure. It will not support combustion. It is ineffective and dangerous as an extinguisher for burning metals, such as aluminium, magnesium or sodium because it accelerates the combustion to the point of explosive violence.

4.2.1 It is common practice to mix it with flammable liquids, such as naphtha, acetone, alcohol to reduce their flammability and flash point. Care is required in the use of such mixtures to make it certain that evaporation has not changed the concentration so that they have actually become flammable.

5. STORAGE AND HANDLING

5.1 Storage

5.1.1 *Corrosion* — If removed from the original containers carbon tetrachloride should be stored in tinned or galvanized iron, lead, monel metal, nickel or especially resistive lined containers. Storage in black iron containers is not recommended and may result in corrosion and leakage of the container.

5.1.1.1 Although not subject to corrosion, aluminium shall not be used in carbon tetrachloride service because under certain conditions it will act as a catalyst.

5.1.2 *Tanks*— If storage tanks are located in an open outdoor area, the vapour and health problems are minimized. Carbon tetrachloride should be stored in a cool place and should be kept dry.

5.1.2.1 For indoor storage, ventilation should be provided at the floor level, as well as in the usual higher locations. Avoid pits, depressions and basements.

5.1.2.2 Each storage tank should have a vent large enough to permit the escape of vapour during filling. Vents from indoor tanks should terminate outdoors, in such location that escaping vapour will not contaminate any workroom. They should be so arranged that vapour will not re-enter the working area. It is to be noted that carbon tetrachloride vapour is 5.32 times heavier than air.

5.1.3 *Drums* — Drums should be kept away from heat and moisture. They should not be kept in the direct rays of sun for a prolonged period. Store with plugs upwards. Each container should be inspected frequently and maintained in good conditions and properly labelled.

5.1.4 *Cans* — Small quantities should be stored in tightly closed, properly labelled containers to avoid leakage, spillage and careless handling as a result of ignorance of the contents.

5.1.5 Storage in glass containers is not recommended unless protected by an outside container.

5.1.6 Uncovered pails or other containers for storing or handling carbon tetrachloride should be prohibited.

5.1.7 *Ventilation* — The workroom should be provided with general ventilation. Since carbon tetrachloride vapour is 5.32 times heavier than air, special mechanical exhaust ventilation, preferably of the down-draft type should be provided in cases where there may be very little air movement, such as storage sheds.

5.1.7.1 Intake to the exhaust ventilating system should be at, or under, the source of the vapour. It should be designed to draw the vapour away from but not past the faces of the operators.

5.1.7.2 For preventing escape of vapour from open tanks, a slot-type lateral intake is useful.

5.1.7.3 If it is used for spraying large parts, closed equipment is recommended.

5.2 Handling

5.2.1 *General* — Each consignment should be unloaded carefully to prevent damage. Each container should be examined carefully for leaks. If any one found, it should be handled with particular care to prevent

further leakage, and removed to a safe place where the leak can be stopped or contents transferred to another container.

5.2.2 *Leaks and Spills* — If leaks or spills occur, only properly protected personnel should remain in the area. Leaking containers should be removed to the outdoor or to a well-ventilated area, and contents transferred to other suitable containers. Rags or mops wet with carbon tetrachloride should be placed in closed containers until they can be dried safely, either outdoors or under ventilation. Clothing wet with carbon tetrachloride should be removed immediately and the body washed to remove any of the carbon tetrachloride which has penetrated. The clothing should not be used again until dry and free of the odour of carbon tetrachloride.

5.2.3 Carbon tetrachloride should as far as possible be handled in closed system.

5.2.4 Devices employing open flames should not be permitted in areas where carbon tetrachloride is used as a solvent.

5.2.5 Where carbon tetrachloride is used in the cleaning of textile fabrics, garments or other materials which provide a large surface area for vaporization of a relatively small volume of the solvent, special care should be taken to provide for effective removal of the vapour by means of suitable hoods, exhaust ducts, or other means to prevent the formation of high concentration of tetrachloride vapour in the air breathed by the operator. The use of open tanks or vessels without such adequate devices should never be permitted.

6. PACKING AND LABELLING

6.1 Packing — Carbon tetrachloride is a hazardous liquid and shall be packed in suitable containers, such as galvanized metal barrels or drums.

6.1.1 For the packing of bulk quantities, tank cars shall be employed.

6.1.2 For the purpose of packing small quantities, small containers such as a tinned can may be used.

6.2 Labelling — Each container (including tank cars) shall carry an identifying label or stencil giving the following information.

a) Name and address of the manufacturer.

b) Quantity, expressed in terms of kilograms or litres.

c) Each container of carbon tetrachloride shall also bear the label given in Fig. 5 of IS : 1260-1958*. The lower half of the label shall have the following words printed in red letters. Any other label or warning or other statement required by statutes,

*Code of symbols for labelling of dangerous goods.

regulations or ordinance may also be used in combination or separately.

CARBON TETRACHLORIDE

DANGER Hazardous vapour and liquid may be fatal if inhaled or shallowed.

Use only with adequate ventilation.

Do not breathe vapour.

Avoid prolonged or repeated contact with skin.

Do not take internally.

7. PREVENTIVE MEASURES

7.0 General — In spite of the toxic nature of carbon tetrachloride if proper measures are taken the hazards involved may be minimized. This depends upon the effectiveness of employee education, proper safety instructions, intelligent supervision and use of safe equipment.

7.1 Employee Selection and Training

7.1.1 *Pripla ement Medical Examination* — Piror to the assignment to the processes involving the handling of carbon tetrachloride, all individuals should have a careful preplacement physical examination and in order to protect the health of the employee, the examining physician may wish to exclude from exposure people with the following conditions.

a) Addiction to excessive use of alcohol, and

b) Liver, kidney or heart diseases.

7.1.2 The education and training of employees to work safely and to use the personal protective equipment or other safeguards provided for them is the responsibility of supervision Training classes for both new and old employees should be conducted periodically to maintain a high degree of safety in handling procedures

7.1.3 Workers should be thoroughly informed of the hazards that may result from improper handling of carbon tetrachloride. They should be cautioned to prevent spills and thoroughly understand that any spills shall be cleaned up immediately. Each employee should know what to do in an emergency and should be Fully informed as to first-aid measures.

7.1.4 Employee education and training should include the following.

a) Instructions and periodic drill or quiz regarding the locations, purpose and use of personal protective equipment.

b) Instruction and periodic drill or quiz regarding the locations of safety showers, eye-baths, bubbler drinking fountains, or the closest source of water for use in emergencies.

c) Instructions to avoid all unnecessary inhalation of vapours of carbon tetrachloride and all direct contact with the liquid.

d) Instructions to report to the proper authority all signs of illness.

e) Instructions to report to the proper authority all cases of equipment failure.

f) Instruction concerning the synergistic effect of alcohol ingestion in this type of toxicity.

7.2 Periodic Health Examination — Employees who are exposed regularly to carbon tetrachloride should have periodic examinations by a physician who is acquainted with the occupational hazard involved. These examinations should include special attention to the kidneys and the liver.

7.3 Personal Protective Equipment

7.3.1 *Availability and Use* — While personal protective equipment is not an adequate substitute for good, safe working conditions, adequate ventilation, and intelligent conduct on the part of employees working with carbon tetrachloride, it is, in many instances, the only practical means of protecting the worker, particularly in emergency situations. One should keep firmly in mind that personal protective equipment protects only the worker wearing it, and other unprotected workers in the area may be exposed to danger.

7.3.1.1 The correct usage of personal protective equipment requires the education of the worker in proper employment of the equipment availble to him.

7.3.1.2 Under conditions which are sufficiently hazardous to require personal protective equipment, its use should be supervised and the type of protective equipment selected should be capable of control over any potential hazard.

7.3.2 *Eye Protection*

7.3.2.1 *Chemical safety goggles* — Whenever there is danger of carbon tetrachloride coming in contact with the eyes and complete protection is required, carefully fitted, cup-type or rubber-framed goggles, equipped with approved impact resistant glass or plastic lenses, should be worn.

7.3.2.2 *Spectacle-type safety goggles* — Whenever continuous eye protection is desirable but complete eye protection is not needed as in laboratories, metal or plastic-rim safety spectacles with unperforated side shields which may be obtained with prescription safety lenses or suitable all-plastic safety goggles, may be used.

7.3.3 *Respiratory Pretection* — Severe exposure to carbon tetrachloride may occur when decontaminating areas following spills, or in case of failure of piping or equipment. Employees who may be subject to such exposure should be provided with proper respiratory protection and trained in its use and care. Available types are described below.

a) *Self-contained breathing apparatus* permitting the wearer to carry a supply of oxygen or air compressed in the cylinder and the self-generating type which produces oxygen chemically. These allow considerable mobility. The length of time a self-contained breathing apparatus provides protection varies according to the amount of air, oxygen, or regenerating material carried. Compressed oxygen should not be used where there is danger of contact with flammable liquids, vapours or sources of ignition, especially in confined spaces, such as tanks or pits

b) *Positive pressure hose masks* supplied by blowers requiring no internal lubrication. The wearer shall be able to use the same route for exit as for entrance and shall take precautions to keep the hose line free of entanglement. The air blower shall be placed in an area free of contaminants.

c) *Air-line masks* supplied with clean, compressed air. These are suitable for use only where conditions will permit safe escape in case of failure of the compressed air supply. These masks are usually supplied with air piped to the area from a compressor. It is extremely important that the air supply is taken from a safe source, and that it is not contaminated bv oil decomposition from inadequate cooling at the compressor. The safer method is to use a separate compressor of the type not requiring internal lubrication. Pressure-reducing and relief valves, as well as suitable traps and filters, shall be installed at all mask stations.

d) *Industrial canister-type gas' masks* equipped with full face pieces for absorbing carbon tetrachloride vapour. These will afford protection against concentrations not exceeding 2 percent by volume when used in accordance with manufacturers' instructions. The oxygen content of the air shall not be less than 16 percent by volume. The masks should be used for relatively short exposure periods only. They may not be suitable for use in an emergency since, at that time, the actual vapour concentration is unknown and an oxygen deficiency may exist The wearer shall be warned to leave the contaminated area immediately on detecting the odour of carbon tetrachloride. This may indicate that the mask is not functioning properly, that the vapour concentration is too high, that the canister is exhausted, or that the mask is not properly fitted.

7.3.4 *Head Protection* — 'Hard' hats should be worn where there JS danger from falling objects. If hard hats are not considered necessary,

soft brimmed hats or caps may be worn to give protection against liquid leaks and splashes.

7.3.5 *Foot Protection* — Leather or rubber safety shoes with built-in steel toe caps are recommended for workers handling drums and cans of carbon tetrachloride. Rubbers may be worn over leather safety shoes. Rubbers and shoes should be thoroughly cleaned and ventilated after contamination.

7.3.6 *Body, Skin and Hand Protection* — Sustained or intermittent skin contact with liquid carbon tetrachloride may produce dermatitis at the site of contact. It is imperative that contaminated clothing be removed promptly and laundered before re-use. Affected areas of the body should be washed thoroughly with soap and water (except the eyes). As a general hygienic measure, facilities for personal cleanliness should be provided and washing before lunch and at the end of the day, should be encouraged. Gloves and aprons of neoprene or other impervious material may be worn to protect the body against carbon tetrachloride splashes. These garments shall be cleaned inside and out each time they are contaminated.

7.4 Air Analysis

7.4.1 For safety in the use of carbon tetrachloride a knowledge of the concentration of its vapour in the work room is essential.

7.4.2 Physical methods using gas interferometer, halide meter or leak detector, infra-red mass spectrograph and gas chromatograph, may be used.

7.4.3 A reliable chemical method for determination of carbon tetrachloride is described in detail in Appendix A.

8. FIRST AID

8.0 General — After exposure to carbon tetrachloride it is important to remove the patient from the contaminated area promptly. In case of contact of the liquid with the skin or eyes immediate flushing with running water is important. In all cases of exposure, severe enough to produce symptoms of illness, call a physician immediately.

8.1 Contact with Skin and Mucous Membranes — In extensive skin contact the patient should get under the shower immediately, if such is available. Clothing and shoes should be removed under the shower. Lanolin or cold cream may be used to restore the oil to the skin which has been defatted by carbon tetrachloride.

8.2 Contact with Eyes — If liquid carbon tetrachloride has entered the eyes, irrigate immediately with water This may be done with an eye bath, if available, a gentle stream of water from a hose or by pouring water from a clean container. The eyelids should be held apart during the

irrigation to ensure contact of water with all the tissues of the surface of the eye and lids. If pain is still present it is permissible as a first aid measure to instill 2 or 3 drops of 0 5 percent pontocaine solution or an equally effective anaesthetic. No oils or oily ointments should be instilled unless ordered by a physician

8.3 Ingestion — If carbon tetrachloride has been swallowed call a physician immediately. Vomiting should be induced by having the patient stick his finger down his throat or by giving large quantities of warm salt water (2 tablespoons to a glass of water). If vomiting occurs, give more water in order to attempt to wash out the stomach. *Do not give anything by mouth to an unconscious patient.*

8.4 Inhalation — Remove patient to an uncontaminated area. If breathing has stopped an effective means of artificial respiration should be started immediately. If oxygen inhalation apparatus is available, oxygen should be administered, but only by a person authorized for such duty by a physician. The patient should be kept warm but not hot.

CAUTION — Persons exposed to carbon tetrachloride should not be given alcohol, oils, fats or epinephrine.

9. CLEANING AND REPAIRS OF TANKS

9.1 Cleaning or making repairs inside a tank may be hazardous even though the tank formerly contained a non-toxic, non-flammable material Work in carbon tetrachloride tanks is particularly hazardous because of the harmful properties of the product. Since it is nonflammable, no precautions are needed to prevent ignition of the vapour.

9.2 The tank or equipment to be repaired should first be emptied of all liquid; and all pipes leading to and from the tank (except vents) after draining should be disconnected or blanked off.

9.2.1 If steaming is necessary, because of the presence of sludge or deposits or tank walls, steam lines should be large enough to raise the tank temperature above the boiling point of the carbon tetrachloride—water mixture and the steaming should be continued until the carbon tetrachloride vapours have been removed. The tank should then be cooled, preferably by filling with water to overflowing, and drained.

9.2.2 The tank should then be purged with fresh air and the air should be tested for carbon tetrachloride vapour and oxygen content by approved methods before permitting personnel to enter. Ventilation should be continued during cleaning, repairing or inspection.

9.3 Entering Tank

9.3.1 No one should enter a tank until it has been tested and found to be safe and a permit secured from an authorized person. Persons entering

the tank should be equipped with self-contained breathing equipment or an air-line respirator.

9.3.2 One man on the outside of the tank should keep the man in the tank under observation and another man should be available nearby to aid in rescue if any of the men in the tank is overcome

9.3.3 Proper equipment, such as life lines, persona! protective equipment, should be provided and used.

9.3.4 A supplied air respirator or self-contained breathing apparatus, together with rescue harness and life line should always be located outside the tank entrance for rescue purposes, regardless of the type of respiratory equipment or air supply which is provided for employees inside the tank.

9.3.5 If the work extends over a considerable period, or is interrupted, frequent and regular tests of vapour concentration should be made to determine whether recontamination has taken place.

9.3.6 In addition to protecting the workmen actually engaged in cleaning and repairing the tank, attention should be paid to the protection of workers in nearby operations.

9.3.7 During the course of the work, frequent tests should be made to determine that the atmosphere in the tank is being maintained within the safe range This precaution is necessary because residues not completely removed by washing may recontaminate the tank atmosphere.

9.3.8 If a tank cleaner or repairman is overcome, he should be rescued and given first-aid treatment immediately.

9.4 Under no circumstances should a rescuer enter a tank to remove a, victim of over-exposure without self-contained breathing equipment, a safety harness and an attached life fine. The free end of the life line should be manned by an attendant located outside the tank. Another attendant should be immediately available to assist in the rescue if needed. The rescuer should be in the view ot the outside attendant at all times or in constant communication with him.

10. WASTE DISPOSAL

10.1 For sake of economy, carbon tetrachloride should be reclaimed from sludge. Distill contaminated degreasing solvent until no condensed carbon tetrachloride flows to the water separator. Steam strip the residue from the solvent stills before discarding.

10.2 Carbon tetrachloride is extremely toxic, insoluble in water, not biologU cally dcgradable, and corrosive to some materials. Hence do not release carbon tetrachloride or its residues to water courses or municipal sewer

without permission from the proper authority, and make sure that materials of construction are appropriate.

10.3 In the presence of an excess of water, or at very high temperatures, such as occur in open flames, carbon tetrachloride decomposes to give hydrochloric acid and other products. Accordingly, do not dump carbon tetrachloride into incinerators, heaters, or boilers. Handle residues in covered receptacles; avoid direct contact, or exposure to vapours from hot residues, and keep away from food and drinking water.

10.4 If regulations do not forbid, smaller quantities of carbon tetrachloride or process residue sludges may be evaporated by pouring on dry ground at a safe distance from buildings etc. Free-flowing sludge that might soak into the ground immediately should first be poured into shallow containers to allow evaporation to take place. Workers disposing of carbon tetrachloride should have adequate protective equipment and should remain upwind from the disposal area.

APPENDIX A

(*Clause* 7.4.3)

DETERMINATION OF CARBON TETRACHLORIDE IN AIR

A-0. GENERAL

A-0.1 The method is based on adsorption of carbon tetrachloride by silica gel, extraction by *iso*propyl alcohol and its hydrolysis by alkali. The conversion to inorganic chloride is complete to 85 percent. The chloride is estimated by titration, with standard silver nitrate solution. Other organic halogen compounds interfere.

A-1. APPARATUS

A-1.1 Silica Gel Adsorber and Suitable Blower

A-1.2 Electric Oven Adjustable to 50°C

A-2. REAGENTS

A-2.1 Silica Gel — Wash with *iso*propyl alcohol, drain in a Buchner funnel, wash several times with water, then dry overnight at 115° to 120°C. Used silica gel is recovered by a similar procedure. After being treated, every batch of silica gel should be tested for chloride by a blank determination.

A-2.2 *Iso*propyl Alcohol — 99 percent, technical grade distilled over potassium hydroxide. Blanks should be run.

A-2.3 Potassium Hydroxide Pellets — chloride-free.

A-2.4 Standard Silver Nitrate Solution — 0 01 N. 1.70 g per litre of water. For precise work, standardize against sodium chloride.

A-2.5 Acetic Acid — 6 N.

A-2.6 Phenolphthalein Indicator Solution

A-2.7 Potassium Chromate Solution — 10 percent.

A-3. COLLECTION OF SAMPLE

A-3.1 Using 20 g of silica gel in the adsorber, sample for 10 to 30 minutes at the rate of 1 to 2 litres per minute. After sampling, transfer silica gel from adsorber to two clean, dry test-tubes, labelling the silica from the inlet arm of the adsorber and that from the outlet arm.

A-4. PROCEDURE

A-4.1 Carry out the analysis on each portion of silica gel obtained from each of inlet and outlet arms of the adsorber separately.

A-4.2 Transfer the silica gel to a dry 50-ml Erlenmeyer flask. Add 25 ml *iso*propyl alcohol, and let stand for 1 hour with occasional shaking. Pipette 10 ml into a 25-ml Erlenmeyer flask, add 4 pellets caustic potash, cork and place overnight in the oven at 50°C. Transfer to 250-ml casserole, washing flask twice with 2-ml portions of water. Add 1 drop phenolphthalein, and 6 N acetic acid drop by drop until the solution is just acidic. Add 6 drops of potassium chromate solution and titrate with standard silver nitrate solution in yellow light. Carry out a blank test also.

A-5. CALCULATION

A-5.1 Carbon tetrachloride, ppm by volume in air $= \frac{v \times 158{\cdot}8 \times 100}{V \times 85}$

where

v = volume in millilitres of silver nitrate solution required for silica in both the arms, and

V = volume in litres of air used for analysis.

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